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4-methylenebut-2-en-4-olide [108-28-1]

Synonyms: 4-methylenebut-2-en-4-olide; cis-4-Methylenebut-2-en-4-olide;
Protoanemonin;

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Formula $C_5H_4O_2$

CAS RN 108-28-1

ACX Number X1016365-4

Density

Refractive Index

Evaporation Rate

Flash Point ($^{\circ}C$)

DOT Number

Comments

Molecular Weight 96.0854

Melting Point ($^{\circ}C$)

Boiling Point ($^{\circ}C$)

Vapor Density

Vapor Pressure

Water Solubility

EPA Code

RTECS

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L1: Entry 3 of 11

File: USPT

Jan 30, 2001

DOCUMENT-IDENTIFIER: US 6179966 B1

TITLE: Method for producing acrylic acid

Brief Summary Text (6):

The acrylic acid-containing aqueous solution contains many by-products, other than acrylic acid, such as acetic acid, formic acid, formaldehyde, furfural, acrolein, acetaldehyde, propionic acid, maleic acid, benzaldehyde, protoanemonin and the like.

Detailed Description Text (4):

Into a 2 m.sup.2 wet-wall type evaporator was fed an acrylic acid-containing aqueous solution, obtained by two-step catalytic vapor phase oxidation of propylene, and containing 55% by weight of acrylic acid, 41% by weight of water, 3% by weight of acetic acid, and each several tens ppm to several thousands ppm of acrolein, formaldehyde, furfural, acetaldehyde, propionic acid, maleic acid, benzaldehyde, protoanemonin and so on as impurities, at a rate of 50 kg/hr, and heated with a saturated steam at 1.1 kg/cm.sup.2. The generated vapor was fed to an azeotropic dehydration column. The system was designed such that the bottom liquid from the wet-wall type evaporator was circulated back to the evaporator with a pump except a part of the bottom liquid, which was withdrawn to outside with a pump. The air was supplied to the bottom of the evaporator at a rate of 0.3% by volume based on the boiled up vapor. Polymerization inhibitors were supplied to the evaporator so that the concentrations of hydroquinone and the concentration of copper dibutyldithiocarbamate were about 2,000 ppm and 100 ppm, respectively.

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L1: Entry 5 of 11

File: USPT

Oct 25, 1994

DOCUMENT-IDENTIFIER: US 5358611 A

TITLE: Method of reducing impurities in aqueous monomer solutions

Brief Summary Text (8):

Japanese patent 81-41614 discloses a method of reducing the level of protoanemonin in acrylic acid by treating either the aqueous acrylic acid solution resulting from the vapor-phase oxidation, the extracted acrylic acid/solvent mixture, or the glacial acrylic acid. The method disclosed therein requires the addition of 0.5% to 1% by weight of the solution to which it is being added of a nitrous acid salt, nitrogen oxide or nitrosobenzene, and a polymerization inhibitor.

Detailed Description Text (8):

To a 300-milliliter four neck flask equipped with a magnetic stirring bar, an air inlet, a thermometer and an exit and a return tube for the recirculation of monomer, was added 60 grams of 28-32 percent by weight aqueous acrylic acid solution, containing from 400-1,000 parts per million ("ppm") of HQ, prepared by vapor phase oxidation of propylene. The protoanemonin (PTA), furfural and benzaldehyde (PhCHO) levels of the aqueous acrylic acid solution were determined by high pressure liquid chromatography (HPLC) and are reported in ppm based on the aqueous monomer solution. The magnetic stirring bar was activated and air was continuously bubbled through the aqueous monomer solution. A 200 Watt, medium pressure, quartz, mercury vapor lamp (available from Ace Glass Co., Vineland, N.J. catalog no. 7825-32) was turned on, and allowed to equilibrate in a photochemical reactor (available from Ace Glass Co., Vineland, N.J. catalog no. 7878). After fifteen minutes, the aqueous acrylic acid solution was continuously pumped through the exit tube into the photochemical reactor and back to the flask through the return tube. An average residence time of 33 seconds was provided by maintaining a constant volume of 20 milliliters of acrylic acid solution in the photochemical reactor and a flow rate of 36.4 milliliters per minute. The levels of furfural, PTA, PhCHO and HQ were measured periodically by HPLC. The temperature of the aqueous acrylic acid solution remained 24.degree. C. The data appear below.

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L1: Entry 8 of 11

File: USPT

Dec 17, 1974

DOCUMENT-IDENTIFIER: US 3855081 A
TITLE: CHEMICAL PROCESS

Abstract Text (1):

Acrylic acid is purified by removal of inter alia protoanemonin by fractional distillation under controlled conditions to recover pure acrylic acid as distillate.

Brief Summary Text (10):

According to a further aspect of the present invention it has been found that an impurity present in the acrylic acid and which is removed by the above described process is the substance protoanemonin (1) having the structure, ##SPC1##

Detailed Description Text (5):

After the stripping of isopropyl acetate solvent in a 30 plate .times. 1 in. I.D. Oldershaw column fitted with a thermosiphon reboiler and reflux divider operating at 12 kN/m.sup.2 pressure, the main product stream contained: acrylic acid, 66.1 percent w/w; acetic acid, 27.0 percent; protoanemonin, 0.04 percent and traces of formaldehyde, propionic acid and isopropyl acetate. The crude acid product stream was distilled in a 1 in. I.D. .times. 45 plate Oldershaw column operating at 5.3 kN/m.sup.2 pressure to give a base product comprising acrylic acid, 91.6 percent w/w; protoanemonin, 0.05 percent and traces of carboxylic acids.

CLAIMS:

1. A method for improving the quality of acrylic acid monomer containing more than 20 ppm of protoanemonin having the structure ##SPC2##

which comprises removing protoanemonin from the monomer by fractional distillation in a column having at least 10 distillation trays and operating with return of reflux to the column, and removing acrylic acid monomer substantially free from protoanemonin as a fraction boiling in the range of about 53.degree. to 57.degree.C at 20-22 mmHg from the upper part of the column.

3. A process as defined in claim 1 wherein the acrylic acid removed from the column contains less than 20 ppm of protoanemonin.

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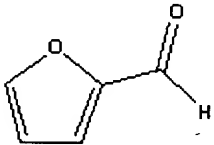
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Search**Furfural [98-01-1]**

Synonyms: 2-Formyl furan; 2-Furaldehyde; 2-furaldehyde; 2-furancarboxal; 2-furancarboxaldehyde; 2-furfural; 2-furylmethanal; alpha-Furfuraldehyde; alpha-furole; artificial ant oil; artificial oil of ants; Fufural; Fural; Furaldehyde; Furfural; Furfural ; Furfuraldehyde; Pyromucic aldehyde; U1199;



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Formula	C ₅ H ₄ O ₂	Molecular Weight	96.0854
CAS RN	98-01-1	Melting Point (°C)	-36.5
ACX Number	X1001098-5	Boiling Point (°C)	167
Density	1.159	Vapor Density	3.3
Refractive Index	1.525	Vapor Pressure	2
Evaporation Rate		Water Solubility	8.3 g/100 mL
Flash Point (°C)	60	EPA Code	U125
DOT Number	UN 1199 Flammable Liquid	RTECS	LT7000000
Comments	Colorless to light brown liquid which darkens in light and air, with an odor like almonds. LIGHT/AIR		

SENSITIVE.
Synthesis of
tetrahydrofuran and
furfuryl alcohol,
phenolic and furan
polymers.

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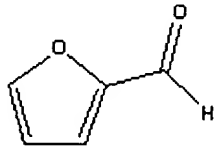
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Search**Furfural [98-01-1]**

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